INTERLABORATORY STUDY 90-6

MERCURY IN REAGENT WATER AND STP EFFLUENT

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MERCURY IN REAGENT WATER AND STP EFFLUENT

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1. SUMMARY AND CONCLUSIONS

This interlaboratory study was initiated, as part of an ongoing program of the Quality Management Office (QMO), Laboratory Services Branch (LSB) of the Ontario Ministry of the Environment (MOE), to evaluate the ability of environmental laboratories to measure mercury in reagent water and in STP effluent.

Thirty five laboratories participated in the study. Four samples in each of the above matrices, to a total of eight, were prepared by the QMO and distributed to the participants. The participants were requested to use their analytical method conforming to the MISA (Municipal and Industrial Strategy for Abatement) regulations. One laboratory reported results for the reagent water samples only.

The results as they were received were entered on to a LOTUS 123® spreadsheet.

The performance of the participants was evaluated using the King-Selliah graphical method[2].

TABLE 1

SUMMARY OF STUDY FINDINGS

# of Participants	35
Acceptable performance (within warning limits)	15 (42.9%)
Out of control/ Erratic	5 (14.3%)
Low bias	8 (22.9%)
High bias	5 (14.3%)
Insufficient sensitivity	1 (2.9%)
Concentration independent (intercept) bias	7 (20.0%)
Concentration dependant (slope) bias	5 (14.3%)

2. RESULTS AND DISCUSSIONS

Each laboratory received a total of eight samples consisting of an unspiked sample, two low spikes and one high spike sample in each of the two matrices. All but one laboratory reported results for all the 8 samples. As expected the majority of the laboratories reported 'not detected' (or less than detection limits) for the unspiked reagent water sample. Hence, the results of this sample were not used in the graphical evaluation. About half the participants reported 'not detected' (or less than detection limits) for the unspiked STP effluent sample. These results too were not not used in this evaluation. From the entire data set the following sub-sets of data were extracted for the King-Selliah[2] graphical evaluation.

a) Sample D2 versus sample D4 (one low and high spike in reagent water). This set of data will be identified in this report as **D24**.

b) Sample D3 versus sample D4 (other low and high spike in reagent water). This set of data will be identified in this report as **D34**.

c) Sample E2 versus sample E4 (one low and high spike in STP effluent). This set of data will be identified in this report as **E24.**

d) Sample E3 versus sample E4 (other low and high spike in STP effluent). This set of data will be identified in this report as **E34.**

Each of the above four data sets was evaluated separately. Accordingly, each pair of results of a laboratory corresponding to the above four sets of data are assessed individually. The outcomes of the four evaluations were then considered in the overall assessment of individual laboratory performance.

Warning and control limits have been used as the basis to flag laboratories that did not demonstrate acceptable repeatability or reproducibility.

The term 'acceptable' throughout these evaluations is based on overall average repeatability of those participants reporting comparable data.

Based on the standard deviations of the 'selected laboratories' (i.e. after rejecting outliers), the evaluation is performed either on absolute scale (concentration units) or on relative scale (as percentage of target or median). The former will result in a rectangular graph and the latter will result in a square graph

In the preliminary run, three of the four sets were automatically evaluated in the relative scale. For purposes of consistency and comparisons of evaluation outcomes between the pairs of data for each laboratory, the fourth set (**D34**) was also evaluated on the relative scale.

Also, as the repeatability (S_w) estimates obtained in these four sets were not significantly different from each other, the individual estimates were pooled and the pooled S_w was used to define the warning and control limits for all four sets.

2.1 SUMMARY OF RESULTS FROM PARTICIPATING LABORATORIES

The results of each of these evaluations are summarized in Tables 2.0 & 2.1. and Figures 2.1 to 2.4. Table 2.0 is the statistical summary of all the evaluations. The "S_w" indicated in these tables represents the average within-laboratory standard deviation (repeatability) estimated for that particular set based on the results of this study as described in appendix I.

The outcome of the evaluation of each laboratory is presented in Table 2.1. The key to this table is as follows:

A	Acceptable performance
ER	Both results erratic
H	Biased high
He	Biased high and/or erratic
HI	Biased high, possible intercept problem
HI	Biased high, probable intercept problem
Hs	Biased high, possible slope problem
HS	Biased high, probable slope problem
IS	Insufficient sensitivity
L	Biased low
Le	Biased low and/or erratic
LI	Biased low, possible intercept problem
LI	Biased low, probable intercept problem
Ls	Biased low, possible slope problem
LS	Biased low, probable slope problem
OC	Out of control-one result erratic
WAI	Warning: Slight imprecision
WHI	Warning: Biased high, probable intercept problem
WHS	Warning: Biased high, probable slope problem
WHs	Warning: Biased high, possible slope problem
WLI	Warning: Biased low, probable intercept problem
WLS	Warning: Biased low, probable slope problem
WLs	Warning: Biased low, possible slope problem
WOC	Warning: Out of control-one result erratic

TABLE 2.0

STATISTICAL SUMMARY

ALL LABORATORIES								
Set	D24		D34		E24		E34	
Sample	D2	D4	D3	D4	E2	E4	E3	E4
# of Labs	35	35	35	35	34	34	34	34
Mean(mg/L)	0.0066	0.0179	0.0076	0.0179	0.0077	0.0221	0.0118	0.0221
Median(mg/L)	0.0003	0.0014	0.0005	0.0014	0.0004	0.0016	0.0006	0.0016
S_{all}(mg/L)	0.03716	0.09785	0.04220	0.09785	0.04283	0.11984	0.06510	0.11984
SELECTED LABORATORIES								
Set	D24		D34		E24		E34	
Sample	D2	D4	D3	D4	E2	E4	E3	E4
# of Labs	22	26	29	26	29	32	31	32
Mean(mg/L)	0.0003	0.0014	0.0005	0.0014	0.0004	0.0016	0.0006	0.0016
Median(mg/L)	0.0003	0.0014	0.0005	0.0014	0.0004	0.0016	0.0006	0.0016
S_{sel}(mg/L)	0.00002	0.00012	0.00009	0.00012	0.00005	0.00025	0.00010	0.00025
REPEATABILITY S_w								
	8.754% (N=29)		6.760% (N=29)		7.175% (N=29)		3.901% (N=28)	
Pooled S_w = 6.895%								

TABLE 2.1
SUMMARY OF OVERALL LABORATORY PERFORMANCE

LAB CODE	D24	D34	E24	E34
3001	A	A	A	A
3002	LI	Le	Li	WLS
3003	A	A	A	A
3004	A	A	A	A
3005	HI	HI	WHs	HI
3006	A	A	A	A
3013	A	A	A	A
3015	IS	IS	IS	IS
3018	A	A	OC	ER
3019	A	OC	A	A
3020	A	A	A	A
3023	A	A	A	A
3027	Li	WLS	WLS	WLS
3028	HI	WHS	WHI	A
3029	A	A	A	A
3032	A	A	A	A
3035	WLs	LS	LS	LS
3037	H	Hi	HS	HS
3038	A	A	A	A
3039	L	Ls	Li	Ls
3040	OC	Le	WOC	WOC
3041	A	A	A	A
3042	A	A	OC	A
3043	H	H	H	H
3044	Hi	Hs	H	HI
3045	A	A	A	A
3047	A	A	A	A
3048	Ls	Ls	OC	LS
3049	A	ER	OC	WHs
3050	A	A	A	A
3051	A	A	A	A
3052	LI	WLI	A	A
3053	LI	WLS	A	WLS
3054	A	HI	A	A
3055	Li	LI		

Thirty five laboratories reported results for all the samples of this study. One laboratory reported results for the reagent water samples only. Fifteen laboratories (42.9% of the participants) produced results that were within the acceptable limits in all the evaluations. These fifteen laboratories are considered as overall good or acceptable performers and are capable of generating valid analytical data. Among the rest three laboratories were flagged in only one of the four evaluations. These laboratories may be considered as moderate performers.

In addition to the fifteen laboratories that produced results within the acceptable limits, two other laboratories, to a total of seventeen (48.6% of participants), produced results within the acceptable limits for the reagent water samples. These two laboratories while demonstrating capability of measuring reagent water samples, experienced difficulty in measuring the more complex matrix (STP effluent) samples. It may be concluded that half the participants of this study are capable of generating valid analytical data in simple matrices such as reagent water.

One laboratory was unable to measure low concentration study samples. The Method Detection Limit (MDL) of this laboratory was at least an order of magnitude higher than those for most participants.

Concentration dependant biases (slope biases) appear to be the major problem for five laboratories that were flagged in this study. Such biases are caused by either inaccurate standards or inadequate calibration procedure. Use of certified reference standards to validate 'in-house' standards or use of the same source of 'quality' external standards by all laboratories will improve the data comparability.

Seven laboratories demonstrated definite intercept dependant biases. Such biases are caused by inappropriate base-line and/or background and/or blank correction.

Four laboratories were flagged as being out of control. These laboratories must achieve greater control over the entire analytical system before diagnosis of biases is possible.

For reagent water samples, the observed mean value of the most comparable laboratories were essentially equal to the target values. Thus it may be concluded that the comparable laboratories on average are accurate in measuring reagent water samples.

The mercury content of the STP effluent was determined to be below the detection\reporting limit by about half of the participants. Therefore the spiked effluent data was not corrected for 'background' in the evaluation. When the background was taken into consideration, the median of the spiked recovered was essentially equal to the spike. Therefore we may conclude that a complex matrix such as STP effluent does not have an adverse effect on the sensitivity of the measurement.

A few laboratories showed evidence of excessive rounding off. These laboratories reported results in one significant figure whereas most participants reported two or more significant figures. This excessive rounding off or lack of sensitivity could have possibly affected their outcomes in these evaluations.

The analytical methodology used by the participants in this study is summarized in Table 2.3. All except one participant used some kind of oxidative acid digestion followed by cold vapour atomic absorption. Nitric acid/sulphuric acid/potassium permanganate/ persuphate digestion, followed by reduction of excess permanganate by hydroxylamine hydrochloride is the popular choice of sample preparation. Majority of the laboratories reported using stannous chloride as a reductant to generate elemental mercury vapour.

2.2 ASSESSMENT OF INDIVIDUAL LABORATORY PERFORMANCE

Laboratory 3001

Acceptable performance

Laboratory 3002

This laboratory is flagged in all four evaluations as being biased low. The intercept dependant bias seen in two sets is masked in the other sets by the imprecision of the data.

Laboratory 3003

Acceptable performance.

Laboratory 3004

Acceptable performance

Laboratory 3005

This laboratory is flagged in all four evaluations as being biased high. Intercept dependant bias seems to be the main problem.

Laboratory 3006

Acceptable performance

Laboratory 3013

Acceptable performance

Laboratory 3015

This laboratory demonstrated lack of sensitivity to measure the study samples.

Laboratory 3018

Although this laboratory performed well with the reagent water sets, it was flagged erratic on the effluent sets. The laboratory is out of control.

Laboratory 3019

Moderate performance. This laboratory is flagged in one of the four sets as being out of control.

Laboratory 3020

Acceptable performance

Laboratory 3023

Acceptable performance

Laboratory 3027

This laboratory is flagged in all four evaluations as being biased low. Concentration dependant bias seems to be the main problem.

Laboratory 3028

This laboratory is flagged in three of the four evaluations as being biased high. Intercept dependant bias seems to be the main problem.

Laboratory 3029

Acceptable performance

Laboratory 3032

Acceptable performance

Laboratory 3035

This laboratory is flagged as showing slope dependant low bias in all four evaluations.

Laboratory 3037

This laboratory is flagged in all four evaluations as showing high bias. Slope dependant bias is very evident with the effluent samples.

Laboratory 3038

Acceptable performance

Laboratory 3039

This laboratory is flagged in all four evaluations as showing low bias. The laboratory is also imprecise.

Laboratory 3040

This laboratory is flagged in all four evaluations. The laboratory is out of control.

Laboratory 3041

Acceptable performance

Laboratory 3042

Moderate performance. This laboratory is flagged in one of the four sets as out of control.

Laboratory 3043

This laboratory is flagged in all four evaluations as showing extremely high bias.

Laboratory 3044

This laboratory is flagged in all four evaluations as showing very high bias. The intercept dependant bias observed in one of the effluent sets may be masked in other sets by the imprecision of the relevant data .

Laboratory 3045

Acceptable performance

Laboratory 3047

Acceptable performance

Laboratory 3048

This laboratory is flagged in all four evaluations. Concentration dependant low bias seems to be the main problem.

Laboratory 3049

This laboratory is flagged in three of the four sets. The laboratory is out of control.

Laboratory 3050

Acceptable performance

Laboratory 3051

Acceptable performance

Laboratory 3052

This laboratory is flagged in reagent water sets. Intercept dependant low bias seems to be the main problem.

Laboratory 3053

This laboratory is flagged in three of the four evaluations. Imprecision of the data precludes any conclusion as to the nature of the bias.

Laboratory 3054

Moderate performance. This laboratory is flagged in one of the all four evaluations as showing intercept dependant high bias.

Laboratory 3055

This laboratory analyzed reagent water samples only. They are flagged in both sets as showing intercept dependant low bias.

3. BIBLIOGRAPHY

1. Ontario Regulation 695/88 under the Environmental Protection Act; Effluent Monitoring General.

2. Classification of Systematic Errors using Two Samples at Different Concentrations, D.E.King & S.S.Selliah, *in preparation.*

3. Graphical Diagnosis of Interlaboratory Test Results, W.J.Youden, Industrial Quality Control, XV, No 11, May 1959.

4. APPENDIX

I. EVALUATION METHODOLOGY

Evaluation of the laboratory performance in this study was performed by an automated spread-sheet and graphical procedure described in the paper "Classification of Systematic Errors using Two Samples at Different Concentrations" by D.E.King and S.S.Selliah[2].

A step-wise summary of this evaluation procedure is given below.

I.1 Summary of The Two-Sample Performance Evaluation Procedure

1. Split two samples of different concentrations among a number of laboratories for analysis/measurement using their current methodology.

2. Enter data on LOTUS 123® spreadsheet.
   Calculate median (L_m,H_m), means and standard deviations for each sample.
   Tabulate data and return to laboratory analyst for verification.
   Correct database if transcriptional errors were reported.

3. Evaluate high sample data:

   i) reject all results which differ from the median (H_m) by more than 10%
   ii) calculate median(H), mean and standard deviation (S_h)
   iii) re-include data if within 3 times S_h
   iv) reiterate ii) and iii) until no further data is included
   v) calculate relative standard deviation of the final selected data (CV_h)

4. Evaluate low sample data:

   i) use 3 x CV_h x median(L_m) to exclude possible outliers
   ii) calculate median(L), mean and standard deviation (S_l)
   iii) reinclude data if within 3 times S_l)
   iv) reiterate ii) and iii) until no further data is included.

5. Determine paired sample performance criteria:

   i) examine the ratio of S_h/S_l and if:
   <2 use data as reported in concentration units
   otherwise convert to % recovery based on expected value if known (otherwise use median values (H,L))
   ii) prepare paired sample scatter diagrams of all data
   iii) calculate perpendicular distance from each point to the two error lines (PD_{slope}, $PD_{intercept}$).
   iv) determine the median (PD_{median}) of all perpendicular distances to the appropriate 45 degree line (intercept error line or slope error line for absolute or relative scale respectively)
   v) calculate the bias for each laboratory (ie the distance along the appropriate 45 degree line) and if:
   <4.5 times PD_{median} select the PD to the 45 degree line
   other wise select the lesser of the two PDs (PD_{slope} or $PD_{intercept}$)
   vi) determine the average of all selected PD values less than 2.5 times the PD_{median} and use this average to estimate the average repeatability S_w (see reference 3).
   vii) set warning limits for repeatability = 2 times S_w
   viii) set control limits for repeatability = 3 times S_w
   ix) set warning limits for possible bias = 3 times S_w
   x) set control limits for possible bias = 4.5 times S_w

6. Code performance based on location of points on the diagram using LOTUS 123® program:

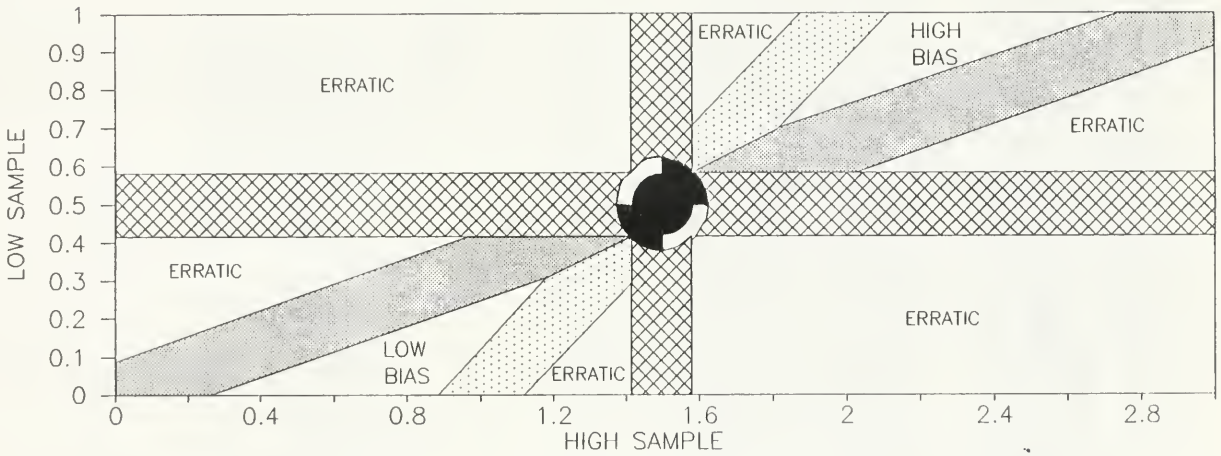
   i) in upper left or lower right quadrant (erratic)
   ii) in lower left or upper right quadrant (biased low or high)
   iii) on horizontal or vertical axis (out of control)
   iv) on diagonal line through origin (slope or standard problems)
   v) on diagonal line not through origin (intercept or blank problems)
   vi) prepare summary table of performance assessment

I.2 Limits for Maximum Interlaboratory Repeatability and Reproducibility

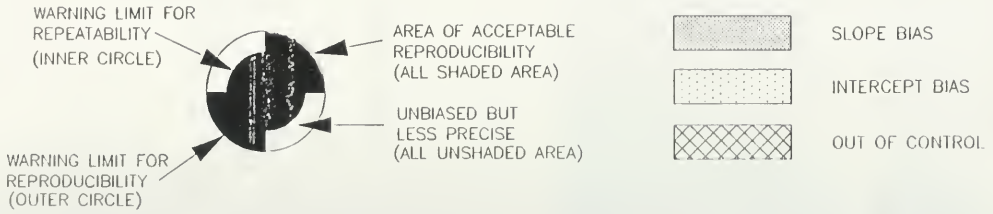
The average perpendicular distance (PD) from the bias lines represents the interlaboratory estimate of within laboratory repeatability. It is used to estimate S_w (see Appendix I.1). Warning limits and control limits for repeatability are set at $2S_w$ and $3S_w$, respectively. Note that the factors used are somewhat arbitrary but they represent approximately 95% and 99% confidence intervals.

Additional tolerance is required for the effect of variability in preparing and using standards on a day to day or among laboratory basis. But the overall estimate of reproducibility includes data from laboratories with excessive bias. In lieu of this S_r is set as a criterion for acceptable reproducibility. S_r is set at 1.5 S_w. Therefore the warning and control limits for reproducibility are set at $3S_w$ and $4.5S_w$ respectively. Based on the f-test a ratio $(S_r/S_w)^2$ exceeding 2.3 (i.e 1.5^2) would be considered significant with a risk of error of less than 10%, 5%, and 1% respectively for 10, 20, and 35 degrees of freedom. Results that exceed warning and control limits determined from this desired maximum interlaboratory (DMI) reproducibility (S_r) are deemed to be possibly or probably biased respectively.

FIGURE 2.0



KEY TO SHADED AREAS



I.3 Two Sample Plot Discussion

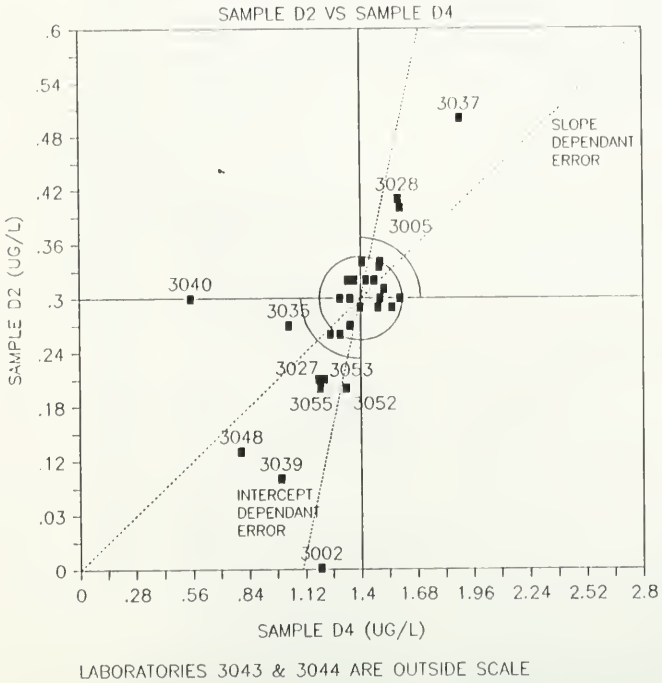
This graphical presentation enables all participants to visualize how they have performed compared to others. The assessment of a laboratory in this study is based on the location of its result on the graph. Figure 2.0 identifies the various regions in a typical graph associated with the different types of problems that might be experienced by the participants. Laboratories with controlled repeatability but showing various degrees of bias will appear in the lower left and upper right quadrants. The two circles drawn in this diagram represent the warning limits for repeatability (S_w) and reproducibility (S_r). Those points within the outer circle but in the upper left and lower right quadrants (not shaded in Figure 2.0) are unbiased but somewhat less precise. Those points within the circle but in the upper right and lower left quadrants are precise but acceptably biased. Thus the area of acceptable performance in this diagram has taken the shape of a keyhole.

In a typical graphical presentation (Figures 2.1 to 2.4), the actual results of each laboratory constitute the points on the graph. The solid lines dividing the graph into four quadrants represent the median results of appropriate samples. The 'keyhole' shaped area of acceptable performance, described earlier, is the area de-limited by the inner circle and the outer arcs in the lower left and upper right quadrants. All laboratories that lie outside this area have exceeded the respective warning limits. The two dotted lines are drawn across the graph representing the slope (concentration) dependant error and the intercept (blank) dependant error. The laboratories that exhibit these types of biases will lie along these lines. All laboratories exceeding warning limits have been identified by their laboratory codes. These laboratories can readily see the nature of their particular problems.

II. GRAPHS & TABLES

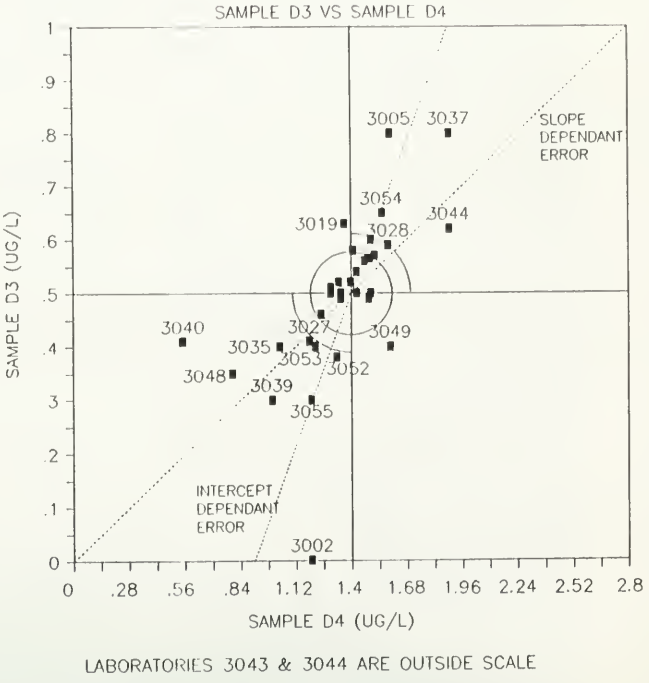
MATRIX: REAGENT WATER

FIGURE 2.1



LABORATORIES 3043 & 3044 ARE OUTSIDE SCALE

FIGURE 2.2



LABORATORIES 3043 & 3044 ARE OUTSIDE SCALE

MATRIX: STP EFFLUENT

FIGURE 2.3

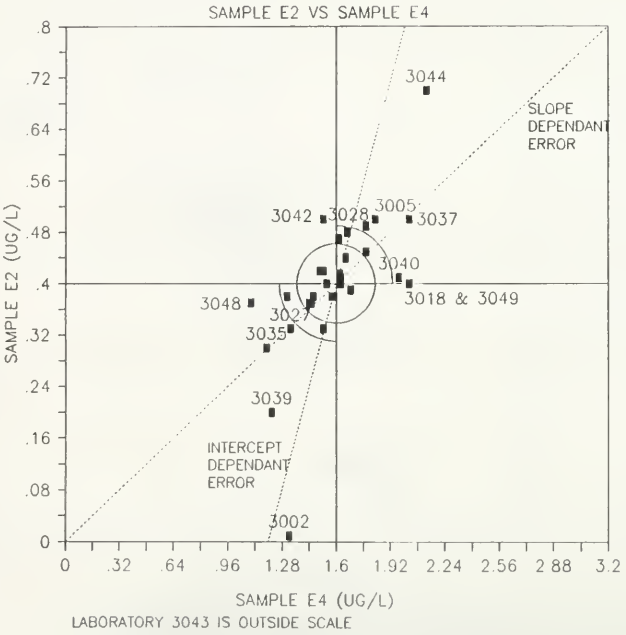


FIGURE 2.4

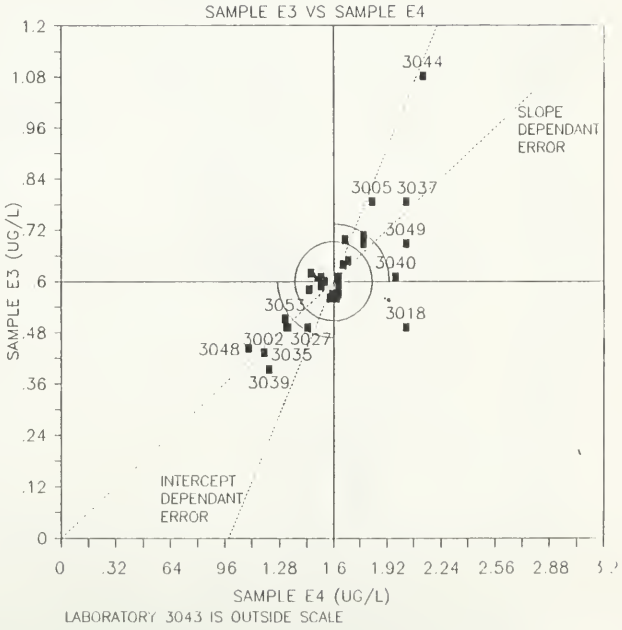


TABLE 2.3
ANALYTICAL METHODOLOGY USED AS REPORTED

LAB CODE	DIGESTION ACIDS	REDUCING AGENT	INSTRUMENTATION
3001	$HNO_3/H_2SO_4/K_2Cr_2O_7/K_2S_2O_8$	$SnCl_2$	COLD VAPOUR AA
3002	$HNO_3/H_2SO_4/KMnO_4/K_2S_2O_8$	$SnCl_2$	COLD VAPOUR AA
3003	ACID-PERMANGANATE		COLD VAPOUR AA
3004	$HNO_3/H_2SO_4/KMnO_4/K_2S_2O_8$	$SnCl_2$	COLD VAPOUR AA
3005	$HNO_3/H_2SO_4/K_2Cr_2O_7/K_2S_2O_8$		COLD VAPOUR AA
3006	$HNO_3/H_2SO_4/KMnO_4/K_2S_2O_8$	$NABH_4$	COLD VAPOUR AA
3013	$HNO_3/H_2SO_4/KMnO_4/K_2S_2O_8$		COLD VAPOUR AA
3015	$HNO_3/H_2SO_4/KMnO_4/K_2S_2O_8$		ICAP-HYDRIDE GENERATION
3018	HNO_3	$SnCl_2$	COLD VAPOUR AA
3019	ACID-PERMANGANATE		COLD VAPOUR AA
3020	$H_2SO_4/KMnO_4/K_2S_2O_8$	$SnCl_2$	COLD VAPOUR -UV
3023	$HNO_3/H_2SO_4/KMnO_4/K_2S_2O_8$		COLD VAPOUR AA
3027	EPA#7470		COLD VAPOUR AA
3028	$H_2SO_4/KMnO_4/K_2S_2O_8$		COLD VAPOUR AA
3029	$H_2SO_4/KMnO_4/K_2S_2O_8$		COLD VAPOUR AA
3032	$ACID/KMnO_4/K_2S_2O_8$	$SnCl_2$	COLD VAPOUR AA
3035	HNO_3	$SnCl_2$	COLD VAPOUR AA
3037	HNO_3/H_2SO_4		COLD VAPOUR AA
3038	$H_2SO_4/K_2Cr_2O_7$/K2S2O8	SnCl2	COLD VAPOUR AA
3039	MOE MC-1		MOE MC-1
3040	$HNO3/K_2Cr_2O_7$	SnCl2	COLD VAPOUR AA
3041	H2SO4/KMnO4		COLD VAPOUR AA
3042	OXIDATION, ACID DIGESTION		COLD VAPOUR AA
3043	MISA REGULAIONS	SnCl2	COLD VAPOUR AA
3044	KMnO4-EPA7470		
3045	STANDARD METHODS		COLD VAPOUR AA
3047	N/A		COLD VAPOUR AA
3048			COLD VAPOUR AA
3049	HNO3/H2SO4/KMnO4/K2S2O8	SnCl2	COLD VAPOUR AA
3050	HNO3/H2SO4/KMnO4		COLD VAPOUR AA
3051	H2SO4/KMnO4/K2S2O8	SnCl2	COLD VAPOUR AA
3052	$KMnO_4$-Continous digestor	SnCl2	COLD VAPOUR AA
3053	HNO3/H2SO4/KMnO4/K2S2O8		COLD VAPOUR AA
3054	HNO3/H2SO4/KMnO4/K2S2O8		COLD VAPOUR AA
3055	OXIDATION, ACID DIGESTION		COLD VAPOUR AA

TABLE 2.4
LIST OF PARTICIPANTS

1	Agri-Service Laboratory
2	ASL Analalytical Service Laboratory Ltd
3	Atomic Energy of Canada
4	Barringer Laboratories Ltd.
5	Beak Analytical Services
6	Bonder Clegg
7	B.C.Research
8	CanTest Ltd.
9	Canviro Analytical Laboratories Ltd.
10	City of Vancouver
11	Clayton Environmental Consultants
12	CPRT Laboratories Inc.
13	EAG Analytical Services
14	Elemental Research
15	Enviroclean
16	Environment Canada
17	Environmental Protection laboratory Inc.
18	Fenwick Laboratories Ltd.
19	Inco Ltd.
20	Lakefield Research
21	Manitoba Environment
22	Mann Testing Laboratories Ltd.
23	Norlab Environmental Services Inc.
24	Novalab
25	Ontario Hydro
26	Ontario Ministry of the Environment,Rexdale
27	Ontario Ministry of the Environment, Thunder Bay
28	Ortech Internatonal
29	Pollutech Ltd.
30	Retek Resource Recovery
31	Technical Service Laboratories
32	Walker Laboratories
33	Williams Operating Corp.
34	XRAL Environmental
35	Zenon Environmental Inc.

TABLE 2.5
RAW DATA

Lab code	D1	D2	D3	D4	E1	E2	E3	E4
3001	.000002	.00034	.00057	.00150	.00005	.00042	.00062	.00160
3002	<.0001	<.0001	<.0001	.0012	<.0001	<.0001	.0005	.0013
3003	<.0001	.00032	.00052	.00134	.0008	.00042	.00062	.0015
3004	<.00002	.00029	.00052	.0014	.00004	.0004	.00062	.0016
3005	.0002	.0004	.0008	.0016	.0002	.0005	.0008	.0018
3006	<.0001	.0003	.0005	.0013	<.0001	.0004	.0006	.0016
3013	<.00005	.0003	.0005	.00135	.0001	.00045	.0007	.00175
3015	<.001	<.001	<.001	.001	<.001	<.001	<.001	.001
3018	.0005	.0003	.0005	.0015	.0002	.0004	.0005	.0020
3019	<.00008	.00032	.00063	.00137	.00013	.00039	.00066	.00166
3020	<.00005	.00031	.00057	.00152	.00006	.00044	.00065	.00163
3023	.00002	.00032	.00056	.00147	.00004	.00040	.00061	.00152
3027	<.00004	.00021	.00041	.00119	.00008	.00033	.0005	.00131
3028	.00007	.00041	.00059	.00159	.00009	.00049	.00072	.00175
3029	<.00005	.00026	.00051	.0013	.00008	.00033	.00062	.0015
3032	<.00017	.00034	.00058	.00141	<.00017	.00047	.00057	.00159
3035	<.00010	.00027	.00040	.00104	<.00010	.00030	.00044	.00117
3037	ND	.0005	.0008	.0019	ND	.0005	.0008	.0020
3038	<.00004	.00032	.00054	.00143	.00010	.00048	.00071	.00164
3039	<.0001	.0001	.0003	.0010	<.0001	.0002	.0004	.0012
3040	.00043	.00030	.00041	.00055	<.00014	.00041	.00062	.00194
3041	<.00008	.00027	0.00049	.00135	<.00008	.00038	.00057	.00155
3042	ND	.0003	.0006	.0015	ND	.0005	.0006	.0015
3043	.16	.22	.25	.58	.10	.25	.38	.70
3044	.00019	.00062	.00062	.0019	.00019	.00070	.0011	.0021
3045	.00005	.00032	.00050	.00143	.00005	.00037	.00059	.00143
3047	<.00010	.00029	.00049	.00149	<.00010	.00038	.00058	.00156
3048	<.00005	.00013	.00035	.00080	<.00005	.00037	.00045	.00108
3049	.0001	.0003	.0004	.0016	.0019	.0004	.0007	.0020
3050	<.00005	.00034	.00050	.0015	<.00005	.00041	.00058	.0016
3051	<.00002	.00026	.00046	.00125	.00007	.00042	.00062	.00148
3052	<.00010	.00020	.00038	.00133	<.00010	.00037	.00050	.00142
3053	<.00010	.00021	.00040	.00122	<.00010	.00038	.00052	.00129
3054	<.00002	.00029	.00065	.00156	.00009	.00038	.00063	.00144
3055	ND	.0002	.0003	.0012	NA	NA	NA	NA